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Research Article

Effect of Open Firing and Electric Furnace Firing on Material Characterization

Erdal Çetintaş[1a]
[1] Ceramics Department, Faculty of Fine Arts, Akdeniz University, Antalya, Türkiye
ecetintas@akdeniz.edu.tr

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ORCID: [a]0000-0002-9814-7574.

Abstract : Ceramic production started with firing earthenware products using fire and has been developed with various techniques and discoveries until today. Although these developments continue, ceramic products produced using traditional methods still exist. Considering today's conditions, it is essential to experimentally examine different firing environments of ceramics, considering both the cost and the sustainability of the products. This study aimed to determine the changes caused by the differences between open firing and electric furnace (800 °C) firing environment in the local pottery production center in Sorkun village of Mihalıççık district of Eskişehir province. For this purpose, water absorption, firing shrinkage, bulk shrinkage, color measurement, chemical analysis (XRF), mineralogical analysis (XRD), differential thermal analysis (DTA) and scanning electron microscopy (SEM) analysis of the samples were performed after two firing process. Based on the obtained results, the values of dry, firing, and bulk shrinkage after open firing suggest that the separation of structural water and/or the complete occurrence of crystal changes in the ceramic sample are not fully achieved compared to electric furnaces. It was determined that whiteness (L^*) and redness (a^*) increased after the electric furnace firing. XRD analysis of results indicated that clinochlore, amphibole, quartz, fluoro-ederite minerals were determined in open firing samples, anorthite, calcium aluminum silicate, magnesiohornblende, quartz minerals were determined in electric furnace firing. The presence of clay minerals in the open firing sample confirms that 800 °C was not fully reached.

Keywords : Ceramic, color, electric furnace, open firing, Sorkun, XRD

1 Introduction

Ceramic production, which started with firing earthenware products using fire, has been developed with various technical solutions and discoveries until today. However, in many ceramic production (pottery) centers located in different geographies, the equipment and tools developed ages ago are used without much change, and this type of production is called "Primitive Pottery" today [1]. Since the beginning of prehistoric research, ceramic production has been one of the most critical indicators in determining social, economic and social differentiation, not only because of technological developments but also because of the form changes they show depending on their functionality [2]. Ceramics are used not only as daily-use items but also as bricks and tiles used in construction, oil lamps, jewelry and ornaments, sarcophagi, and children's toys [3].

The ceramic production cycle consists of raw materials, mud processing, shaping, drying, and firing techniques [4]. Although the clay structure used in ceramic production, shaping techniques and firing methods have changed depending on technological advances, it is known that traditional methods are still maintained. In ancient times, the centers of the ceramic industry were located near clay-rich regions, and the bodies of ceramic pieces were generally prepared from raw materials rich in clay, kaolin, quartz, feldspar, or micaceous formations [5]. In some regions, clay deposits used in antiquity are still used today according to the oldest traditions [6].

Firing is the most critical stage in ceramic production after raw materials', shaping, and drying. The prepared ceramic products' firing process is carried out to gain durability by chemical reactions thanks to heat energy after the drying process. When the studies on firing are examined, it is observed that firing started with open firing, single-chamber firing, firing chamber, and wood-fired kilns, and many alternative firing techniques and kiln (furnace) are used today depending on technological developments [7], [8].

Open firing is an outdoor firing method in which ceramic products are placed side by side and on top of each other, and the fuels (organic materials such as straw, stalks, wood, etc.) are placed in the gaps at the bottom top and between the ceramics and are fired [9]. Since it is open firing, paying attention to weather conditions is necessary. Exposure to wind and contact between ceramic forms and fuel make it difficult to control the atmosphere and the temperature in open firing [10]. Due to the uncontrolled temperature, firing, and color differences are possible [11]. Since the fire in this firing method is in direct contact with the ceramic body, fractures, and cracks can be high during firing. To partially reduce this damage, some regions have been protected the pottery underneath from sudden heat change by stacking previously broken pottery pieces on the upper side of the kiln [12]. In addition to traditional firing methods, electric furnace are widely used to fire ceramic products. Electric furnace are generally square or rectangular fixed kilns. Such furnaces are preferred to easily adjust the maximum temperature, heating rate, and thermal homogeneity.

Since open firing and electric furnace methods affect many properties of ceramic products, the thermal profile of firing including atmospheric conditions (oxydant and reducing atmosphere) can also be considered as a primary factor of ceramic properties. The parameters that characterize the firing thermal profile are maximum temperature, heating rate, heating time, thermal homogeneity, and continuity [13]. The studies have reported that the differences between the thermal profiles of open firings and electrical furnaces are of opposite nature for ceramic production. In kiln firing, the high maximum temperature, low heating rate, and the ability to soaking/firing time are controlled isothermally are controlled, while these properties in open firing are completely variable and uncontrollable [14]–[16]. The thermal profile of open firing cannot be kept constant due to atmospheric conditions. Depending on all these parameters, knowing the reactions that occur during the firing process in ceramic bodies can help determine the firing temperatures.

Considering the previous literature, although firing temperature prediction studies based on phase characterization have been investigated in many studies separately, studies in which the differences in firing environments which are examined together with the structural and phase characterization of the same type of raw materials are a few. This study made inferences about the physical and structural differences that occur after the open firing, which is still traditionally practiced in Sorkun Village and the firing of the same raw material in an electric furnace. In addition, the color change, mineralogical properties, and structural findings obtained after different firing processes were interpreted and discussed.

2 Experimental Methods

This study examined potter's clay and firing in Sorkun Village, one of the important centers of pottery production. Sorkun Village is located 12 km away from the Mihaliççik district of Eskişehir and has a high altitude. The most important source of livelihood of the village is pottery. Two clay mixtures, "red" and "oily soil," are used in pottery making (Figure 1). Ceramic pots in different forms are prepared using mud prepared from these clays and left to dry.

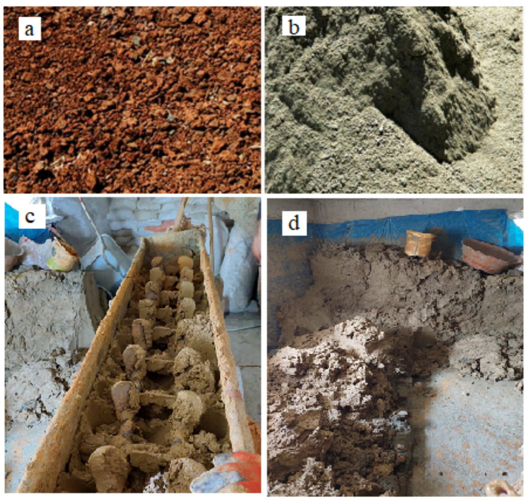

Figure 1: Clay and mud preparation a) red clay, b)oily clay, c) mud mixing device, d) prepared mud.

The open firing process is carried out in an open field outside the village of Sorkun when the wind blows strong from one direction, and ceramic products are fired in one batch (300 to 500 pieces). First, on the straw and sawdust dust laid on the ground in the open field, the ceramic products are lined up with their mouths directed to the ground. A row of firewood and pots are laid and the firewood is lined up in the direction of the wind and burned. It is stated in the literature that the temperature value reaches approximately 750-800 ° during firing [17]. At the end of the firing process, the pots are pulled with a "çeykel" and left to cool (Figure 2).

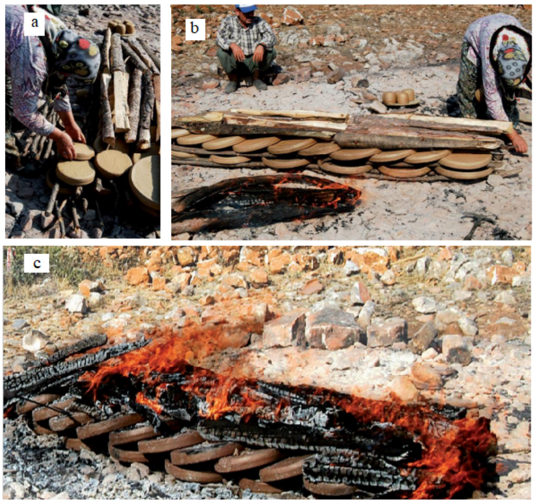

Figure 2: Open firing process in Sorkun [17].

Since this study aims to determine the differences between open firing and electric kiln firing, a sample of the product obtained from open firing in Sorkun village was taken. The clay obtained from Sorkun village was shaped 30x5x1 cm for the electric furnace. After drying, 800 °C firing was carried out in the electric furnace (Figure 3).

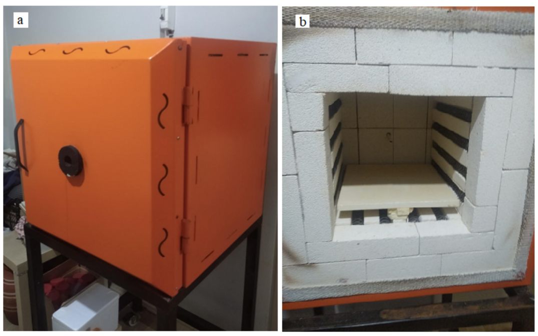

Figure 3: Electric furnace a) View of the furnace b) Inside furnace.

Physical tests such as water absorption, firing shrinkage, bulk shrinkage, and color measurements were performed to examine the changes in ceramic forms in different firing environments. The water absorption, firing shrinkage, bulk shrinkage tests in Table 1 were carried out according to the test methods explained in the section of "Experiments Applied to Clay and Kaolin" in "Ceramic Technology" book written by Arcasoy [18]. Color measurements were performed using a Konica Minolta color meter based on the CIELAB system. L, a, and b color measurements were recorded after open and electric firing. Elemental determination XRF examinations of the ceramic samples used in the study for the characterization tests were carried out on a Rigaku brand device. Semi-quantitative chemical analysis results of the prepared samples regarding weight percent were obtained. Samples taken from different firing environments were ground to a grain size of 100 μm, and XRD analysis was performed with a the Shimadzu brand XRD-6000 model device. The samples were scanned at 2 °/min and analyzed in the range of 2°-70° (2Θ) ganiometer diffraction angle and 2000 cps (intensity) peak intensity. Additionally, thermal analysis (DTA/TG) of the raw clay sample was analyzed by SETARAM / labSys Evo device. Scanning electron microscope (SEM) examinations, which are necessary to examine the surface morphology of the ceramic samples and to perform microchemical analysis of the minerals, were carried out on the LEO brand, model 1431-VP SEM at AKU Technology Application Research Center (TUAM).

3 Results and Discussion

The changes occurring in different firing environments after open firing, which has traditionally existed in Eskişehir Province Sorkun Village, and electric furnace applied in today's conditions were examined. The findings and discussions are given in this section.

The water absorption, dry shrinkage, firing shrinkage, and bulk shrinkage of the samples used in this study are presented in Table 1. It was found that the water absorption value for the open firing sample is 15.28%, whereas it is 14.30% for the electric kiln sample. The amount of water absorption is directly proportional to the amount of pores present in the body. According to these results, the water absorption rate is high in the open firing sample due to the number of pores present in the body. When the values obtained from dry shrinkage, firing shrinkage and bulk shrinkage tests are examined, the values are 5.44%, 0.47% and 5.89% in the open firing sample, while these values are 6.22%, 3.02%, and 9.27% respectively in the electric kiln sample. Based on the obtained results, the values of dry, firing, and bulk shrinkage after open firing suggest that the separation of structural water and/or the complete occurrence of crystal changes in the ceramic sample are not fully achieved compared to other firing conditions.

Table 1: Water Absorption, Dry Shrinkage, Firing Shrinkage, and Bulk Shrinkage values.

Firing Types	Water Absorption (%)	Dry Shrinkage (%)	Firing Shrinkage (%)	Bulk Shrinkage (%)
Open Firing	15.28	5.44	0.47	5.89
Electric Furnace	14.30	6.22	3.02	9.27

The color parameters of the ceramic samples after open firing and electric kiln firing are given in Table 2. Among the parameters used in color measurement, the L^* value is in the range of black-white (0-100), the a^* value shows red-green, and the b^* value shows a yellow-blue scale.

Table 2: Color parameter values obtained from ceramic samples.

Color Parameters	Open Firing	Electric Furnace
L	51.54	53.80
a	9.63	15.13
b	18.06	23.26

L parameter of the color properties expresses lightness. While the L value was 51.54 after open firing, the L value was measured as 53.8 after electric furnace firing. Accordingly, it was determined that the L value increased in the electric furnace firing. The change in the a parameter indicates redness when $a > 0$ and greenness when $a < 0$. When Figure 4 is examined, it is seen that the value of open firing has a value of 9.63 and the value of electric furnace firing has a value of 15.13. According to this, it was determined that the color changed towards redness in electric firing. The b parameter of the color properties expresses yellowness and blueness ($b > 0$ yellow and $b < 0$ blue). When the b parameter is examined, it is seen that it has a value of $b > 0$ after both firing. Accordingly, the change with the highest b value was obtained after electric furnace firing.

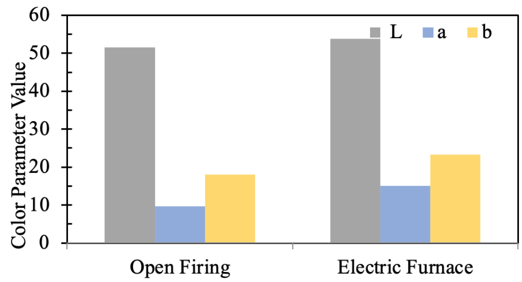

Figure 4: Color change in different firing environments.

In general, it is known that the color of ceramic products is mainly due to the minerals (especially iron oxide) and their amounts in the clay that make up the material and that the firing environment conditions are also adequate [19]–[21]. The terms oxidation and reduction refer to how much oxygen is in the kiln's atmosphere while the kiln is firing. An oxidation atmosphere has plenty of oxygen for the fuel to burn. A reduction atmosphere occurs when the amount of available oxygen is reduced. This may not sound like things that will affect your pottery, but it can. The reduction process, when oxygen is leeched out of your kiln atmosphere and pottery, can change the texture or color of clay. In the study, although the ceramic clay was the same in both firings, an increase in color parameters was observed after electric kiln firing. It is thought that this difference in color arises due to the inability to achieve a uniform temperature during the firing process. When clays are fired at high temperatures, the color increases towards dark red. In other words, it shows that the temperature is high in electric kiln firing, but the open firing temperature cannot reach the electric firing temperature.

The chemical analysis results of the ceramic samples after firing are presented in Table 3. Upon examining the obtained chemical results, it was found that SiO2 is the highest chemical component by weight in the samples. The SiO2 content of the open hearth firing sample is 46.33%, whereas it is 48.56% in the electric kiln firing sample. Considering the MgO and CaO ratios, which indicate the presence of dolomite and calcite, which are known as carbonates in ceramic samples, it was determined that the MgO ratio was approximately 16.30% in both firing samples. In comparison, the CaO ratio was 9.05% in the open firing and 6.07% in the electric furnace firing. One of the clays that make up the Sorkun mud was named red. It indicates the presence of hematite minerals (Fe_2O_3) in this clay. The F_2O_3 ratios after firing are very close to each other and vary between 11.56% and 11.63%.

Table 3: XRF results of ceramic samples.

Chemical Composition (% by weight)	Firing Types	
	Open Firing	Electric Furnace
SiO_2	46.33	48.56
Al_2O_3	13.30	13.82
Fe_2O_3	11.56	11.63
MgO	16.30	16.32
CaO	9.05	6.07
K_2O	1.08	0.99
Na_2O	0.64	0.66
TiO_2	1.03	1.13
Cr_2O_3	0.19	0.19
MnO	0.22	0.24
P_2O_5	0.17	0.26
NiO	0.13	0.13

According to XRD results, clinochlore, amphibole, quartz, fluoro-ederite minerals were detected in the ceramic sample in open firing; anorthite, magnesiohornblende and quartz minerals were detected in the sample during electric furnace firing. Clinochlore is generally a product of hydrothermal alteration of amphibole, pyroxene and biotite and is associated with serpentine, calcite, dolomite etc. As a result of open firing XRD analysis, the presence of clay minerals (clinochlore, amphibole) is seen in the structure and shows that very high temperatures for sintering could not be reached.

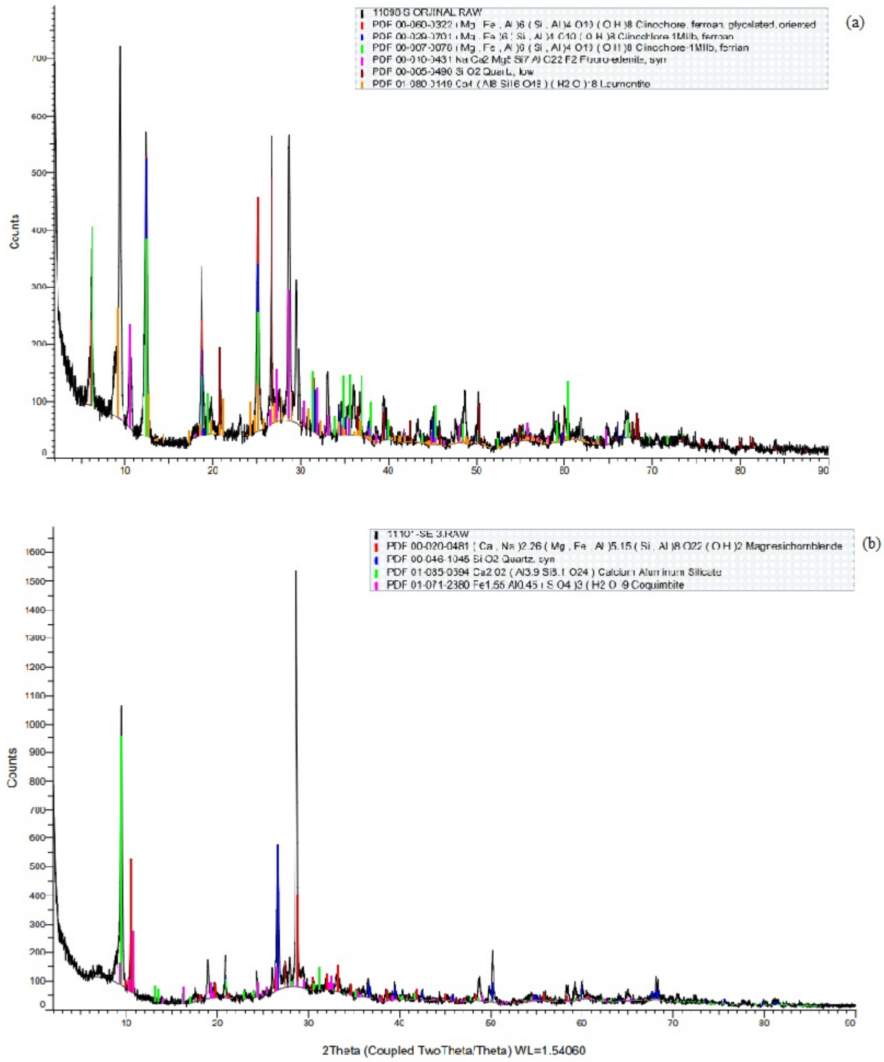

Figure 5: XRD patterns of ceramic a) Open firing (I: illite, In:Indialite, M: Montmorillonite, A: Albite, Q: Quartz,H: Hematite), b) Electric furnace firing (I: illite, In:Indialite, A:Albite, Q: Quartz).

To interpret the structural changes in the sample due to temperature variations, the DTA-TG results of the raw clay sample are presented in Figure 6. The characteristic endothermic and exothermic reactions identified from the TG, DTG (the derivative of the weight loss curve), and DTA curves are indicated on the graphs. The sample exhibits a total weight loss of approximately 10.00% within the temperature range of ∼30-900 °C. In the temperature range of 30-200 °C, the endothermic reaction observed at 110 °C on the DTA curve is attributed to the loss of physical water from the clay minerals. The exothermic reaction at 350 °C observed on the DTA curve within the temperature range of ∼200-400 °C is due to the combustion of likely organics present in the structure. The weight loss in the temperature range of 400-600 °C and the endothermic reaction at 500 °C observed on the DTA curve are associated with the loss of crystal water from the clay minerals. The endothermic reaction observed at ∼650 °C on the DTA curve within the temperature range of ∼600-700 °C is thought to be due to the decomposition of the magnesite mineral. The endothermic reaction observed at ∼740 °C on the DTA curve within the temperature range of ∼700-800 °C is attributed to the decomposition of the calcite mineral.

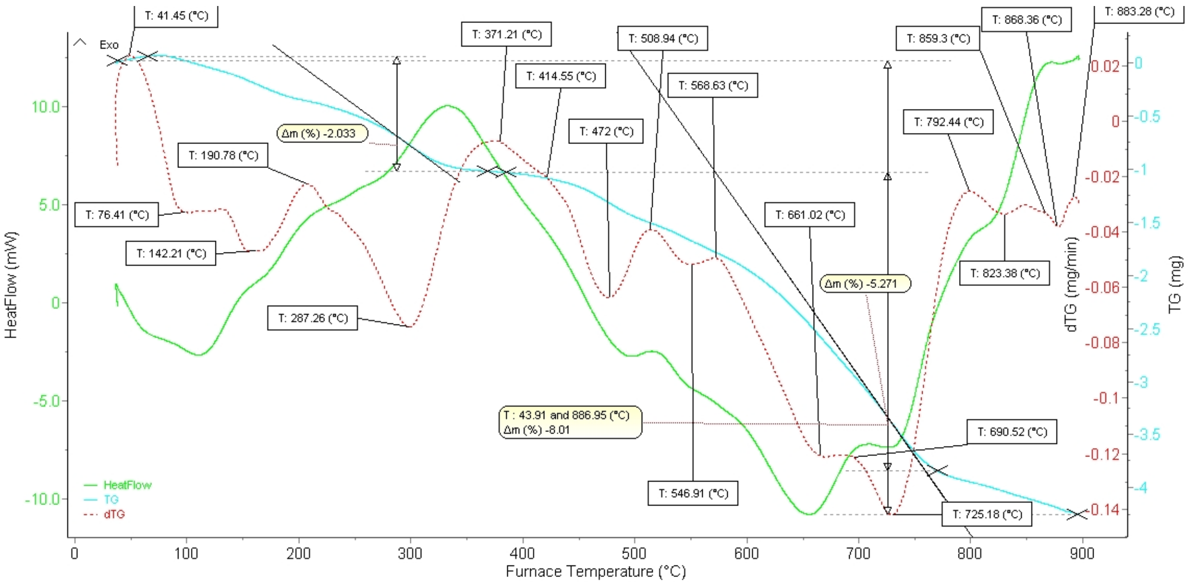

Figure 6: DTA-TG curves of raw clay sample.

Scanning electron microscopy (SEM) was used to examine the microstructure of ceramic samples fired in different firing atmospheres. When the structures of the open firing and electric firing samples are examined in Figure 6, rod-like structures, traces of mineral weathering, and small voids are observed. Since the firing temperature of the electric furnace was 800°C, similar structures are present on the surfaces of the open firing sample and the microstructural properties have not changed much. When the SEM images and EDX analyses are evaluated together with the XRD analysis results obtained, 19.31% $_2O_3$ in the mineral ratios in the rod-like structure in the open firing confirms that it is hematite (Figure 7).

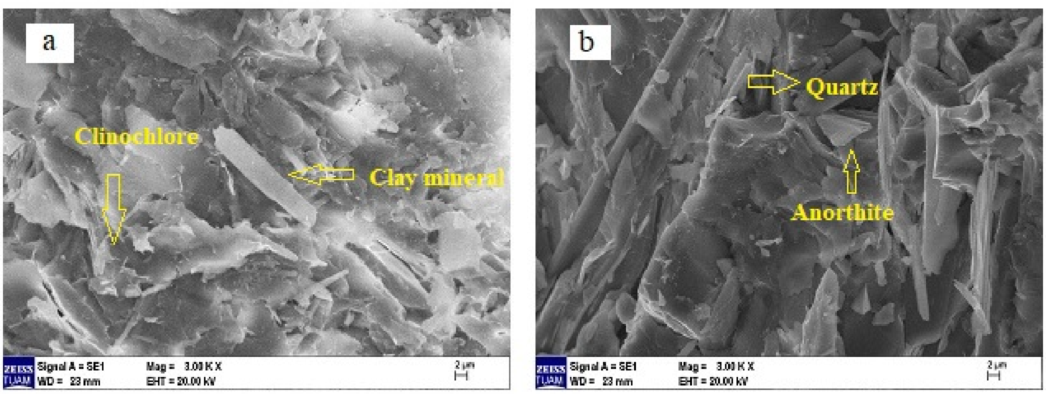

Figure 7: SEM micrographs of firing ceramic samples.

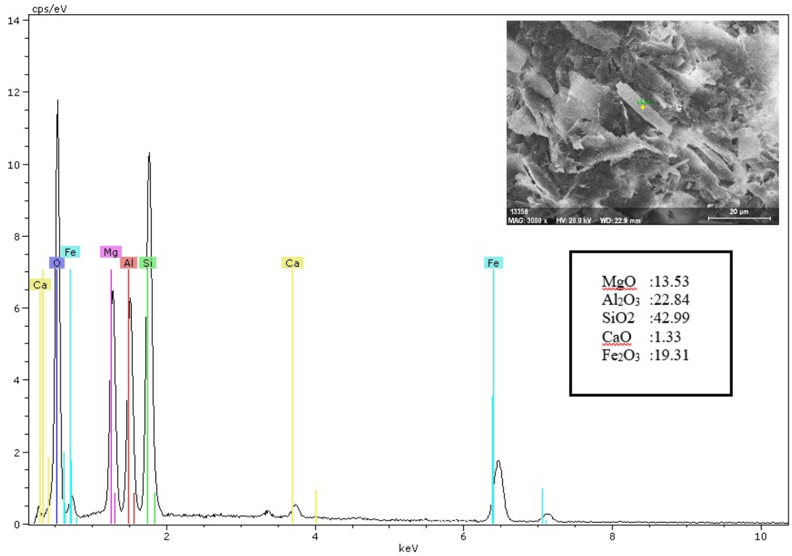

Figure 8: SEM image and EDX analyses of open firing.

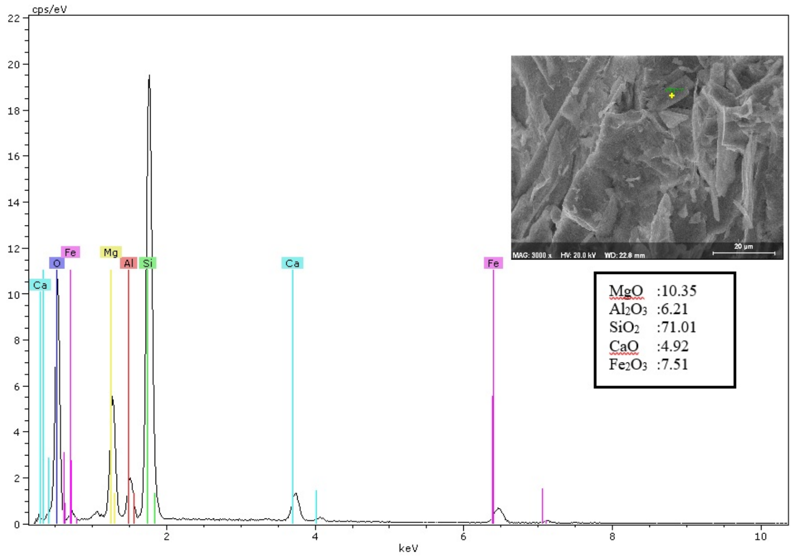

Figure 9: SEM image and EDX analyses of electric furnace firing.

4 Conclusions

Firing of ceramic products made from clay using conventional fire obtained from raw materials has evolved over time with various techniques and discoveries. However, different firing techniques and kilns are used in many ceramic production centers in different geographies today. In today's conditions, it is essential to improve the firing conditions considering both the cost and the sustainability of the products. In this study, the performances of open firing and electric furnace firing were investigated in a local ceramic production place, Sorkun village of Mihalıççık District of Eskişehir.

Accordingly, the water absorption value was found to be higher after open firing. Similarly, dry shrinkage, cooking shrinkage, and bulk shrinkage values were also determined to be higher than those of electric furnace firing. The results obtained show that the separation of crystal water and/or crystal change in the structure of the ceramic sample does not occur completely compared to other firing environments. As a result of the color measurement test, while the L value was 51.54 after open firing, the L value was 53.8 after electric furnace firing. Similarly, it was determined that the color parameters a and b increased in electric furnace firing. It was determined that the chemical analysis values obtained after the firing processes of the ceramic samples were very close to each other. The highest chemical composition by weight after different firing processes of the ceramic samples was found to be SiO_2. Both XRD and SEM-EDX analysis results confirmed this high value. According to the XRD results, clinochlore, amphibole, quartz, fluoro-ederite minerals in the ceramic sample under open firing; anorthite, magnesiohornblende and quartz minerals were detected in the sample under electric furnace firing. As a result of open firing XRD analysis, the presence of clay minerals (clinochlore, amphibole) in the structure shows that very high temperatures are not reached for sintering.

In summary, this research will provide significant benefits in terms of issues such as energy consumption, production speed and sustainability in new studies by bringing together the advanced conditions of the local and industrial environment and determining the changes in different firing environments.

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Authors' Contributions

EÇ designed the structure, carried out the experiments work, the theoretical calculations and wrote up the article.

Competing Interests

The author declares that no inferences have been made.

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